

Partial Translation of JP-A-2003-105095

[Claims]

[Claim 1]

A producing method for a polyamide resin composition obtained by melting and blending two or more polyamide resins, wherein, among the two or more polyamide resins, at least one or more polyamide resins contain an inorganic phosphor compound of from 0.001 to 0.5 parts by weight as phosphor atoms with respect to 100 parts by weight of total of the two or more polyamide resins, and, in melting and blending the two or more polyamide resins, at least one selected from an inorganic acid salt and a hydroxide of a metal of group 1 or 2 of the periodic table is made present in an amount of from 1 to 50 mole equivalents with respect to the total phosphor atoms of the inorganic phosphor compound and an amount of 200×10^{-4} mol/100g or less with respect to total of the two or more polyamide resins.

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[0005]

[Means for Solving the Problems]

The gist of the present invention is a producing method for a polyamide resin composition obtained by melting and blending two or more polyamide resins, wherein,

among the two or more polyamide resins, at least one or more polyamide resins contain an inorganic phosphor compound of from 0.001 to 0.5 parts by weight as phosphor atoms with respect to 100 parts by weight of total of the two or more polyamide resins, and, in melting and blending the two or more polyamide resins, at least one selected from an inorganic acid salt and a hydroxide of a metal of group 1 or 2 of the periodic table is made present in an amount of from 1 to 50 mole equivalents with respect to the total phosphor atoms of the inorganic phosphor compound and an amount of 200×10^{-4} mol/100g or less with respect to total of the two or more polyamide resins.

[0006]

[Embodiments of the Invention]

The polyamide resin to be employed in the present invention is a polyamide resin obtained by a polycondensation of a lactam of normally 4- or larger-membered ring structure or an ω -amino acid, or a polycondensation of a dibasic acid and a diamine. In order to realize a high heat resistance, a high strength and a high rigidity, necessary as a substitute for a metal, a polyamide resin having 20 or less methylene groups, particularly 12 or less methylene groups, in a repeating unit of the polymer, is advantageous. Examples of lactam of 4- or larger-membered ring structure, for use as a raw

material of polycondensation, include ϵ -caprolactam and ω -lauro lactam, examples of ω -amino acid include ϵ -caproic acid, ω -aminocaprylic acid, and ω -aminolauric acid, examples of the dibasic acid include glutaric acid, adipic acid, azelaic acid, sebacic acid, suberic acid, dodecane diacid, isophthalic acid (I), and terephthalic acid (T), and examples of diamine include tetramethylenediamine, nonanediamine, hexamethylenediamine, octamethylenediamine, diaminobutane, m-xylylenediamine (MXDA), and p-xylylenediamine.

[0007]

Specific examples of such polyamide resin include polyamide 4, polyamide 6, polyamide 11, polyamide 12, polyamide 46, polyamide 66, polyamide 610, polyamide 612, polyhexamethylene terephthalamide (polyamide 6T), polyhexamethylene isophthalamide (polyamide 6I), poly-m-xylylene adipamide (polyamide MXD6), poly-m-xylylene dodecamide, and polyamide 9T.

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[0029]

Examples 1 - 4, Comparative Examples 1 - 2, and Reference Example 1

In these Examples 1 - 4 and Comparative Examples 1 - 2, the polyamide resin composition employed 3 components of polyamide MXD6 (A1, A2) produced utilizing an inorganic

phosphor compound for preventing coloration, PA6 (A4), and PA66 (A5). In Reference Example 1, the polyamide resin composition employed same 3 components, except for employing polyamide MXD6 (A3) produced without utilizing the inorganic phosphor compound. In any of these Examples 1 to 4, Comparative Examples 1 to 2, and Reference Example 1, three peaks were observed as melting peak temperatures (Tmp1, Tmp2, Tmp3) at an initial heating, and two peaks (ATmp1, ATmp2) were observed at a re-heating. Also in any, one peak was observed as a crystallization temperature. In Examples 1 to 4 of the present invention, by the use of inorganic acid salt or hydroxide as the component (c), the range of change of initial melting peak temperatures (Tmp1 - 3) and re-melting peak temperature (Tmp1, 2) become smaller in comparison with Comparative Examples 1, 2, not utilizing the component (c), and is comparable to that in Reference Example 1. Also the crystallization temperature (Tcp) is maintained as high as in Reference Example 1, and the re-melting temperatures (Tmp1, 2) are higher than in Comparative Examples 1, 2 and close to those in Reference Example 1. Based on the foregoing, it is found that the resin composition is not influenced easily by a staying, and is almost free from an unnecessary amide exchange reaction and almost free from a loss in the crystallinity. Reference Example 1, not utilizing the inorganic phosphor

compound in the preparation of the polyamide resin, is almost free from a loss in crystallinity and from a change in the melting peaks, but the raw material polyamide resin shows a significant coloration as described above, because the inorganic phosphor compound is not used, thus being of low value as an industrial product and generally not usable for the manufacture of resin composition as a product.

[0030]

[Table 1]

		Ref.Ex 1	Ex. 1	Ex. 2	Ex. 3	Ex. 4	Comp. Ex. 1	Comp. Ex. 2
glass fiber		100	100	100	100	100	100	100
talc		2	2	2	2	2	2	2
polyamide A1	parts by wt.		47				47	
polyamide A2	parts by wt.			47	47	47		47
polyamide A3	parts by wt.	47						
PA6	parts by wt.	42	42	42	42	42	42	42
PA66	parts by wt.	11	11	11	11	11	11	11
NaH ₂ PO ₂ ·H ₂ O	x10 ⁻² pts by wt.	0	6	2.7	2.7	2.7	6	2.7
	x10 ⁻⁴ mol/100g	0	5.7	2.5	2.5	2.5	5.7	2.5
Ca(OH) ₂	parts by wt.	0	0.4	0.1	0.2	0	0	0
	x10 ⁻⁴ mol/100g	0	54	13.6	27	0	0	0
Na ₂ CO ₃	parts by wt.	0	0	0	0	0.3	0	0
	x10 ⁻⁴ mol/100g	0	0	0	0	28.4	0	0
temp. decrease (°C)	Tcp	174	170	169	174	162	160	159
initial heating (°C)	Tmp1	225	225	225	225	224	222	225
	Tmp2	239	235	239	239	239	233	239
	Tmp3	262	260	261	262	262	258	262
re-heating (°C)	ATmp1	211	210	207	210	209	202	206
	ATmp2	225	227	223	228	224	216	222

[0031]

Examples 5 - 7, Comparative Examples 3 - 5, and

Reference Example 2

Table 2 shows polyamide mixtures of 4 components of MXD6 (A1, A2), PA6 (A4), PA66 (A5) and PA12 (A6). Among these, Reference Example 2 is an example utilizing a polyamide MXD6 (A3) produced without utilizing the inorganic phosphor compound. In any of these Examples 5 - 7, Comparative Examples 3 - 5, and Reference Example 2, three peaks (Tmp1, Tmp2, Tmp3) were observed as the melting peak temperatures at the initial heating, and, at the re-heating, Example 6 provided three peaks (ATmp1 - ATmp3). In other Examples, Comparative Examples and Reference Example, two peaks were observed (ATmp1 and 3: the peaks having temperatures close to those in Example 6 were named same as in Example 6 (ATmp1, 3)). Also for the crystallization temperature, two peaks (Tcpl, 2) were observed in each case, except for Comparative Example 4. In a comparison of Examples 5 - 7 with Comparative Examples 3, 4, in Examples 5 - 7 utilizing the inorganic acid salt or hydroxide as the component (c), a range of change between the initial melting peak temperatures (Tmp1 - 3) and the re-melting peak temperatures (ATmp1 - 3) is smaller in comparison with Comparative Examples 3 and 4 not utilizing the component (c), and is comparable to that in Reference Example 2. Also the crystallization temperatures (Tcpl, 2) are maintained as high as in

Reference Example 2, and higher than in Comparative Examples 3, 4. Based on the foregoing, it is found that the resin compositions of Examples 5 - 7 are not influenced easily by a staying, and are almost free from an unnecessary amide exchange reaction and almost free from a loss in the crystallinity. Comparative Example 5, which is a case containing the component (c) exceeding the range of the present invention, may be considered as being not easily influenced by staying but are inferior to Examples in performances, because of a large loss in the tensile strength. Reference Example 2, not utilizing the inorganic phosphor compound in the preparation of the polyamide resin, is almost free from a loss in crystallinity and from a change in the melting peaks, but the raw material polyamide resin shows a significant coloration as described above, because the inorganic phosphor compound is not used, thus being of low value as an industrial product and generally not usable for the manufacture of resin composition as a product.

[0032]

[Table 2]

		Ref.Ex 2	Ex. 5	Ex. 6	Ex. 7	Comp. Ex. 3	Comp. Ex. 4	Comp. Ex. 5
glass fiber		100	100	100	100	100	100	100
talc		2	2	2	2	2	2	2
polyamide A1	parts by wt.				48		48	48
polyamide A2	parts by wt.		48	48		48		
polyamide A3	parts by wt.	48						
PA6	parts by wt.	30	30	30	30	30	30	30
PA66	parts by wt.	11	11	11	11	11	11	11
PA12	parts by wt.	11	11	11	11	11	11	11
NaH ₂ PO ₂ ·H ₂ O	x10 ⁻² pts by wt.	0	2.5	2.5	6.2	2.5	6.2	0.12
	x10 ⁻⁴ mol/100g	0	2.5	2.6	5.8	2.6	5.8	10.8
Ca(OH) ₂	parts by wt.	0	0.1	0.2	0.4	0	0	2
	x10 ⁻⁴ mol/100g	0	13.6	27	54	0	0	270
temperature decrease (°C)	Tcp1	145	145	149	149	145	145	149
	Tcp2	178	170	178	177	159	-	178
initial heating (°C)	Tmp1	175	175	175	175	174	175	175
	Tmp2	235	235	234	233	234	232	233
	Tmp3	257	257	256	255	257	254	255
re-heating (°C)	ATmp1	174	176	178	176	174	174	178
	ATmp2	-	-	208	-	-	-	-
	ATmp3	220	219	228	224	215	205	229
tensile strength	MPa	25.1	22.8	23.1	21.7	24.9	25.0	19.0

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METHOD FOR MANUFACTURING POLYAMIDE RESIN COMPOSITION

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Abstract of **JP2003105095**

PROBLEM TO BE SOLVED: To provide a method for stably manufacturing a desired polyamide resin composition in obtaining the polyamide resin composition from melt-kneading two or more polyamides. **SOLUTION:** A method for manufacturing a polyamide resin composition obtained from melt-kneading two or more polyamide resins is the one wherein at least one or more of the two or more polyamide resins comprise 0.001-0.5 pts.wt. of an inorganic phosphorus compound as a phosphorus atom to 100 pts.wt. of the two or more polyamide resins in total, and when melt-kneading the two or more polyamide resins, at least one selected from inorganic acid salts of the first or second family metals in the periodic table of elements is made to exist in 1-50 mole equivalents to the total phosphorus atoms of the inorganic phosphorus compound and in not more than 200×10^{-4} mol/100 g to the two or more polyamide resins in total.

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Partial Translation of JP-A-49-53945

2. CLAIMS

A gellation preventing method for a polyamide molten substance, characterized in blending, to a polyamide, (A) at least one phosphor compound selected from a phosphinic acid compound (1), a phosphonous acid compound (2), a phosphonic acid compound (3) and a phosphorous acid compound (4) represented by general formulas:

chemical formula (1)

chemical formula (2)

chemical formula (3)

chemical formula (4)

(wherein R_1 to R_7 each represents a hydrogen, an alkyl group, an aryl group, or a cycloalkyl group; and X_1 to X_5 each represents any of groups R_1 to R_7 or an alkali metal; provided that one each of X_1 to X_5 and R_1 to R_7 in each formula may be bonded each other to form a cyclic structure) in an amount in terms of phosphor of 50 to 1,000 ppm by weight with respect to the polyamide, (B) an alkali compound of a general formula:

$Z-OR_8$ (B)

(wherein Z represents an alkali metal; R_8 represents a hydrogen, an alkyl group, an aryl group, a cycloalkyl group or $-C(=O)OZ'$, in which Z' represents a hydrogen or

an alkali metal), as an amount of all the alkali metals in the system, in an amount 1 to 5 times the amount of the phosphor compound (A), and (C) o-phenylenediamine and/or a derivative thereof in an amount of 0.01 to 2 wt% with respect to polyamide.

*** *** *** *** ***

Examples of the polyamide to be employed in the present invention include diamine-dicarboxylic acid condensed polyamide such as polyhexamethylene adipamide, polyhexamethylene sebacamide, polyxylylene adipamide, polyxylylene sebacamide, polyhexamethylene isophthalamide, poly-p-xylylene dodecamide, and polycyclohexanebismethylene dodecamide, and a copolymer and/or a blend utilizing a monomer/nylon salt constituting them.

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